Sandwell Beak Research Group
Energy and Environmental Science

DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUIFACTION

FINAL REPORT

BY

SANDWELL BEAK RESEARCH GROUP

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MICROMEDIA

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OT THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

REFERENCE TESTS USING CLVB ONLY (NO COAL)

	TEST #	2	3	5	20	21	31	32	34	35	41	48	49	73	86
R															
E															
A	Coal Type	NONE													
C	Solvent	CLVB													
T	S/C, wt ratio	-	-	-	-	-	-	-	-	-	-	-	-	-	-
O	Gas Mixture	H2	H2	H2/H2S	H2/H2S	H2	W2/H2S	N2	CO/H2	CO/H2/H2S	CO/H2	CO/H2/H2S	CO/H2	H2	H2/H2S
R	H_2S %, m.a.f. coal	-	-	15	10	0	15	0	0	10	0	10	0	0	15
	CO/H_2, volume ratio	-	-	-	-	-	-	-	2/1	2/1	2/1	2/1	2/1	-	-
C	H_2O, % m.a.f. coal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
O	Catalyst, type	NONE													
N	Pressure, MPa	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
D	Temperature °C	425	425	425	400	400	425	425	425	425	425	425	425	425	425
I	Time, minutes	60	60	60	60	60	60	60	120	120	120	60	60	60	60
T	Coal Drying	-	-	-	-	-	-	-	-	-	-	-	-	-	-
I															
O															
N															
S															
	INPUT														
M	Solvent	259.7	226.2	262.7	295.4	291.7	304.0	297.6	284.4	297.5	288.5	299.1	293.8	277.7	287.9
A	Coal m.a.f	-	-	-	-	-	-	-	-	-	-	-	-	-	-
T	Ash	-	-	-	-	-	-	-	-	-	-	-	-	-	-
E	H_2O	-	-	-	-	-	-	-	-	-	-	-	-	-	-
R	H_2	10.2	9.8	9.3	9.8	9.8	-	-	3.5	3.3	3.4	3.3	3.3	10.4	10.4
I	H_2S	-	-	10.6	5.5	-	10.6	-	-	5.5	-	5.5	-	-	9.2
A	CO	-	-	-	-	-	-	-	99.1	94.7	97.2	94.7	94.7	-	-
L	N_2	-	-	-	-	-	155.0	151.0	-	-	-	-	-	-	-
S	Catalyst	-	-	-	-	-	-	-	-	-	-	-	-	-	-
A	OUTPUT														
L	H_2O	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.3	0.0	0.0
A	Org. Lights	72.8	84.0	67.0	28.1	24.0	96.5	99.6	97.0	129.7	104.3	122.7	101.5	81.6	90.5
N	Heavy	169.9	130.9	170.8	258.6	252.0	67.5	133.4	107.7	77.6	116.3	131.7	141.1	149.2	157.8
C	Washings	*	*	*	*	*	12.6	7.2	35.1	25.1	17.7	6.3	11.7	16.3	20.0
E	Reactor solids	-	-	-	-	-	73.7	-	-	-	-	-	-	-	-
	H_2	8.8	6.9	7.6	8.6	9.8	0.0	0.0	1.8	1.2	2.2	1.8	2.4	9.3	9.3
S.	CO_2	0.1	0.0	1.1	0.2	0.0	1.4	0.0	6.7	10.2	5.9	6.6	4.1	0.2	0.2
	CO	0.0	0.0	1.7	0.0	0.0	0.6	0.0	94.0	91.2	91.9	78.6	87.0	0.0	0.0
	H_2S	5.8	4.6	18.9	8.4	3.5	11.6	0.0	3.3	3.8	2.2	5.8	2.0	4.3	12.3
	C1	3.7	2.8	4.6	1.3	1.2	6.4	5.8	6.6	8.8	6.0	5.6	4.6	3.8	4.3
	C2	3.1	2.4	3.7	1.2	1.2	5.4	4.8	5.2	6.3	5.0	4.9	4.0	3.4	3.6
	C3 C4	5.7	4.4	7.2	3.2	2.7	13.2	10.3	9.3	19.2	15.6	17.7	15.4	8.1	8.4
	N_2	-	-	-	-	-	156.0	150.3	-	-	-	-	-	-	-
	Mat. Bal. %	98.7	94.2	96.5	99.6	97.6	94.7	91.7	94.7	93.0	94.3	94.8	95.7	95.9	99.6
R	IOR (g.)	11.5	10.7	8.4	3.8	3.08	79.0	37.8	27.1	22.2	24.8	18.4	19.1	5.7	9.3
E	Distillable (g.)	n.d.	149.4	165.3	155.0	121.5	~~n.d.~~	~~n.d.~~	154.1	167.3	154.3	179.3	169.1	160.9	174.1
S	C_1-C4 (g.)	12.5	9.6	15.5	5.7	5.1	25.0	20.9	21.1	34.3	26.6	28.2	24.0	15.3	16.3
U	H_2 consumed (gr)	(1.4)	(2.9)	(1.6)	(1.2)	0.0	0.0	0.0	1.7	2.1	(1.2)	(1.5)	(0.9)	(1.1)	(1.1)
L	H_2S (g.)	5.8	4.6	8.3	2.9	3.5	1.0	0.0	0.0	(1.7)	2.2	0.3	2.0	4.3	3.1
T	Hexane Insol. Liq. (g.)	n.d.	37.8	41.7	57.7	65.5	19.5	30.7	37.0	28.7	40.9	44.0	45.7	51.7	52.0
S	S in hex ins.	n.d.	2.7	2.9	4.6	4.8	2.0	2.0	4.0	3.3	2.5	2.7	3.0	3.5	3.8
	H_2O (g.)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.3	0.0	0.0
	Coversion, % m.a.f. coal	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Distillate yield, % total m.a.f.	n.d.	66.0	62.9	~~n.d.~~ 52.5	~~n.d.~~ 41.7	~~n.d.~~ 51.0	~~n.d.~~ 55	54.2	56.2	53.5	59.9	57.6	57.9	60.5

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OT THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS USING ANTHRACENE OIL AS SOLVENT

	TEST #	18	19	46	47	76
REACTION CONDITIONS	Coal Type	F	F	F	F	F
	Solvent	H.A.O.	H.A.O.	A.O.	A.O.	A.O.
	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1
	Gas Mixture	H2	H2	H2/H2S	H2	H2/H2S
	H_2S %, m.a.f. coal	-	-	10	-	10
	CO/H_2, volume ratio	-	-	-	-	-
	H_2O, % m.a.f. coal	-	-	-	-	-
	Catalyst, type	-	-	-	-	-
	Pressure, MPa	17.25	17.25	17.25	17.25	17.25
	Temperature °C	425	425	425	425	425
	Time, minutes	60	60	60	60	60
	Coal Drying	OD	OD	OD	OD	OD
MATERIAL BALANCE	INPUT					
	Solvent	222.5	232.4	244.3	240.8	249.5
	Coal m.a.f	55.6	58.2	61.1	60.2	62.3
	Ash	5.0	5.0	5.4	5.4	5.6
	H_2O	0.0	0.0	0.0	0.0	0.0
	H_2	9.8	9.8	10.1	10.4	10.6
	H_2S	0.0	0.0	5.5	0.0	5.6
	CO	0.0	0.0	0.0	0.0	0.0
	N_2	0.0	0.0	0.0	0.0	0.0
	Catalyst	0.0	0.0	0.0	0.0	0.0
	OUTPUT					
	H_2O	0.0	0.0	0.0	0.0	6.2
	Org. Lights	32.2	36.0	101.2	37.3	42.8
	Heavy	223.4	243.0	157.5	248.4	248.0
	Washings	0.0	0.0	20.7	14.3	6.3
	Reactor solids					
	H_2	9.1	9.1	8.1	8.7	8.9
	CO_2	4.2	4.3	4.8	4.7	4.2
	CO	0.7	0.7	0.8	0.7	0.9
	H_2S	0.1	0.1	4.6	0.0	4.6
	C_1	1.0	1.4	2.0	1.7	1.8
	C_2	0.9	1.1	1.3	1.2	1.5
	C_3 C_4	2.0	1.6	4.0	4.3	3.5
	N_2	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	93.4	97.3	93.2	101.4	98.6
RESULTS	IOR (g.)	9.3	9.9	17.0	23.8	6.2
	Distillable (g.)	207.7	231.3	227.2	236.5	246.9
	C_1-C_4 (g.)	3.9	4.1	7.3	7.3	6.8
	H_2 consumed (gr)	(0.7)	(0.7)	(2.0)	(1.7)	(1.7)
	H_2S (g.)	0.1	0.1	(0.9)	0.0	(1.0)
	Hexane Insol. Liq. (g.)	76.0	34.7	34.6	71.5	31.5
	S in hex ins.	0.4	0.2	0.5	0.4	.35
	H_2O (g.)	0.0	0.0	0.0	0.0	6.2
	Coversion, % m.a.f. coal	83.3	83.0	72.2	60.5	90.0
	Distillate yield, % total m.a.f.	74.7	79.6	74.5	78.6	79.2

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OT THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS CONDUCTED USING FORESTBURG COAL

	TEST #	6	10	11	12	16	17	22	23	24	28	50	51	52	53
R															
E															
A	Coal Type	F	F	F	F	F	F	F	F	F	F	F	F	F	F
C	Solvent	CLYB													
T	S/C, wt ratio	2/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
O	Gas Mixture	H2/H2S	H2	H2/H2S	H2	H2	H2/H2S	H2/H2S	H2	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S
R	H_2S %, m.a.f. coal	15	0.0	10	0.0	0.0	10	10	0.0	15	10	20	40	15	15
	CO/H_2, volume ratio	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C	H_2O, % m.a.f. coal	-	-	-	-	-	-	-	-	-	-	-	-	-	-
O	Catalyst, type	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N	Pressure, MPa	17.2	17.2	17.2	17.2	11.0	11.0	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
D	Temperature °C	425	425	425	425	425	425	400	400	425	425	425	425	425	425
I	Time, minutes	60	60	60	60	60	60	60	60	60	30	60	60	10	20
T	Coal Drying	OD	SD	SD	SD	OD	OD	OD	OD	OD	OD	OD	OD	SD	SD
I															
O															
N															
S															
	INPUT														
M	Solvent	184.1	176.5	188.7	232.6	233.4	249.9	227.0	234.2	236.4	239.2	228.2	233.3	237.4	248.6
A	Coal m.a.f	67.8	43.3	47.3	59.5	58.5	62.6	56.9	58.7	59.2	60.0	57.0	58.3	59.3	62.2
T	Ash	6.1	3.9	4.1	5.3	5.1	5.4	4.9	5.1	5.1	5.2	5.1	5.2	5.3	5.5
E	H_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
R	H_2	9.6	9.8	9.4	9.8	5.3	5.1	9.4	10.4	9.8	9.5	9.4	9.1	9.7	9.7
I	H_2S	10.6	0.0	5.5	0.0	0.0	5.9	5.5	0.0	10.6	5.9	11.4	24.5	8.9	8.9
A	CO	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L	N_2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
B	Catalyst	-	-	-	-	-	-	-	-	-	-	-	-	-	-
A	OUTPUT														
L	H_2O	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
A	Org. Lights	58.7	49.5	55.4	63.1	60.3	61.4	38.6	35.2	70.2	62.1	87.0	112.0	61.0	76.5
N	Heavy	126.3	135.8	127.3	174.6	155.5	191.9	195.6	213.0	157.4	173.2	146.2	11.0	176.2	190.4
C	Washings	35.4	0.0	28.2	16.7	20.2	30.1	39.0	34.0	37.7	42.7	22.2	149.1	41.0	17.8
E	Reactor solids	0.0	0.0	0.0	0.0	0.0	0.0	0.0	-	-	-	-	-	-	-
	H_2	6.0	7.8	6.7	7.8	3.8	2.7	8.1	8.1	5.4	7.0	5.9	5.9	8.1	7.5
g.	CO_2	8.8	5.4	4.5	7.1	5.6	5.6	4.5	5.0	5.2	4.9	3.7	5.5	4.8	5.2
	CO	5.8	0.6	1.8	0.8	0.3	0.7	0.7	0.8	2.1	1.1	0.9	1.4	0.6	0.6
	H_2S	19.3	7.5	12.4	9.9	4.4	10.3	10.0	3.8	19.6	10.1	14.4	19.6	10.0	12.4
	C_1	4.5	3.5	4.3	4.6	4.5	4.3	1.9	1.7	4.2	3.5	5.5	8.0	3.1	3.7
	C_2	3.7	4.3	3.7	5.6	4.0	4.2	1.6	1.6	4.3	2.6	4.1	5.4	2.4	3.1
	C_3 C_4	8.1	9.0	8.0	11.9	5.6	7.9	3.6	5.2	9.3	5.4	11.0	12.8	10.1	10.6
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	99.4	95.7	99.0	98.3	87.4	97.0	100.0	100.0	98.2	97.7	96.7	100.1	99.0	98.0
R	IOR (g.)	16.4	21.6	13.8	27.1	39.0	41.5	20.7	28.4	14.3	24.9	18.8	20.7	29.2	23.2
E	Distillable (g.)	135.8	112.1	129.7	147.8	n.d.	n.d.	131.2	123.4	166.8	n.d.	173.9	169.1	144.3	154.2
S	C_1-C_4 (g.)	16.3	16.8	16.0	22.1	14.1	16.4	7.1	8.5	17.8	11.5	20.6	26.2	15.6	17.4
U	H_2 consumed (gr)	(3.6)	(2.0)	(2.7)	(2.0)	(1.5)	(2.4)	(1.3)	(2.3)	(4.4)	(2.5)	(3.5)	(3.2)	(1.6)	(2.2)
L	H_2S (g.)	8.7	7.5	6.9	9.9	4.4	4.4	4.5	3.8	9.0	4.2	3.0	(4.9)	1.1	3.5
T	Hexane Insol. Liq. (g.)	51.4	45.5	49.6	65.0	52.6	67.2	59.8	57.3	58.4	60.5	55.3	54.8	66.8	66.2
S	S in hex ins.	2.1	1.8	2.2	2.9	2.3	3.2	2.8	2.5	2.4	2.6	2.2	2.2	3.3	3.0
	H_2O (g.)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Coversion, % m.a.f. coal	75.8	50.1	70.8	54.5	33.3	33.7	63.6	51.6	75.8	58.5	67.0	64.5	50.8	62.7
	Distillate yield, % total m.a.f.	53.9	51.0	55.0	50.6	n.d.	n.d.	46.2	42.1	56.4	n.d.	61.0	58.0	48.6	49.6

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS CONDUCTED USING FORESTBURG COAL

	TEST #	54	55	56	57	58	59	59R	60	55R	58R	87	87R
R E A C T O R C O N D I T I O N S	Coal Type	F	F	F	F	F	F	F	F	F	F	F	F
	Solvent	CLVB											
	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
	Gas Mixture	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S
	H_2S %, m.a.f. coal	15	15	20	10	15	0	0	20	15	15	15	15
	CO/H_2, volume ratio	-	-	-	-	-	-	-	-	-	-	-	-
	H_2O, % m.a.f. coal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Catalyst, type	NONE											
	Pressure, MPa	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.2	17.2	17.2	17.2
	Temperature °C	425	400	400	375	375	375	375	375	400	375	375	375
	Time, minutes	40	60	60	60	70	60	60	60	60	70	60	60
	Coal Drying	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
MATERIAL BALANCE	INPUT												
	Solvent	221.1	233.8	225.3	239.9	235.2	238.2	233.2	226.2	229.1	232.8	239.4	237.6
	Coal m.a.f	55.3	58.4	56.3	60.0	59.0	59.5	58.3	56.6	57.3	58.2	59.8	59.6
	Ash	4.9	5.2	5.0	5.3	5.3	5.3	6.3	5.1	6.1	6.3	5.3	5.3
	H_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	H_2	9.7	10.6	10.1	11.4	11.2	11.7	11.8	10.3	10.6	11.4	11.4	11.2
	H_2S	8.9	8.9	11.8	5.8	8.8	0.0	0.0	11.5	9.0	9.1	9.0	8.9
	CO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Catalyst	-	-	-	-	-	-	-	-	-	-	-	-
	OUTPUT												
	H_2O	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.4	4.1	2.6	6.0	4.4	6.6
	Org. Lights	85.2	66.2	53.6	28.5	35.0	27.1	14.6	20.6	36.0	15.0	8.8	14.5
	Heavy	149.0	174.6	172.9	226.6	219.3	243.3	259.2	227.4	230.8	257.2	245.8	251.3
	Washings	12.5	38.4	39.5	40.0	24.4	17.5	11.2	19.4	11.6	14.8	41.0	26.3
	Reactor solids	-	-	-	-	-	-	-	-	-	-	-	-
	H_2	6.2	7.5	7.8	9.1	9.3	9.9	11.5	8.8	8.7	10.6	10.3	10.4
	CO_2	4.2	3.9	3.8	4.3	3.2	4.2	3.5	3.3	3.9	3.4	3.4	3.5
	CO	0.8	0.5	0.5	0.4	0.5	0.2	0.0	0.4	0.4	0.1	0.0	0.0
	H_2S	10.4	11.1	12.9	6.7	9.8	2.2	0.9	12.4	10.3	9.5	9.3	8.9
	C_1	4.6	2.3	2.3	0.9	1.1	0.9	0.3	1.0	1.4	0.4	0.4	0.3
	C_2	3.7	2.0	1.9	0.8	0.9	0.8	0.2	0.8	1.2	0.4	0.3	0.3
	C_3 C_4	11.4	7.9	7.5	5.5	1.6	3.3	2.3	1.5	4.9	0.0	2.0	0.0
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	96.3	99.2	98.1	100.1	95.5	98.3	99.5	96.8	99.9	99.9	100.2	99.9
RESULTS	IOR (g.)	16.6	14.4	17.0	29.9	23.7	40.7	44.03	24.8	20.3	28.5	35.1	36.6
	Distillable (g.)	152.1	163.3	134.5	98.8	95.4	94.1	57.8	76.1	112.6	76.2	63.2	70.8
	C_1-C_4 (g.)	19.7	12.2	11.7	7.2	3.6	5.0	2.8	3.3	7.5	0.8	2.7	0.6
	H_2 consumed (gr)	(3.5)	(3.1)	(2.3)	(2.3)	(1.9)	(1.8)	(0.3)	(1.5)	(1.9)	(0.8)	(1.1)	(0.8)
	H_2S (g.)	1.5	2.2	1.1	0.9	1.0	2.2	0.9	0.9	1.3	0.4	0.3	0.0
	Hexane Insol. Liq. (g.)	59.7	60.7	56.5	58.5	69.7	46.1	51.4	69.1	61.7	61.5	59.6	67.6
	S in hex ins.	2.6	2.5	2.5	2.4	3.1	2.0	n.d.	3.2	n.d.	n.d.	2.4	3.4
	H_2O (g.)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.4	4.1	2.6	6.0	4.5	6.6
	Coversion, % m.a.f. coal	69.9	75.3	69.8	50.2	59.8	31.6	24.5	56.2	64.5	51.0	41.3	40.0
	Distillate yield, % total m.a.f.	55.0	55.7	47.8	32.9	32.4	31.6	19.8	26.9	39.3	26.1	21.1	23.8

Sandwell Beak Research Group
Energy and Environmental Science

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OT THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS CONDUCTED USING FORESTBURG COAL

	TEST #	61	62	63	64	77	78	82	83	65	65R	66	67	68	88	89	94
R																	
E																	
A	Coal Type	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F
C	Solvent	CLVB															
T	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
O	Gas Mixture	H_2/H_2S	H_2/H_2S	H_2/H_2S	H_2	H_2	H_2/H_2S	H_2	H_2/H_2S	H_2/CO	H_2/CO	$H_2/CO/H_2S$	$H_2/CO/H_2S$	H_2/CO	H_2/CO	$H_2/CO/H_2S$	$H_2/CO/H_2S$
R	H_2S %, m.a.f. coal	10	15	20	0	0	15	-	15	0	0	15	15	0	0	15	15
	CO/H_2, volume ratio	-	-	-	-	-	-	-	-	1/24	1/24	1/24	2/1	2/1	1/1	1/1	1/1
C	H_2O, % m.a.f. coal	-	-	-	-	-	-	-	-	0.0	0.0	0.0	0.0	0.0	50.0	50.0	0.0
O	Catalyst, type	NONE															
N	Pressure, MPa	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
D	Temperature °C	450	450	450	450	450	450	450	450	400	400	400	400	400	400	400	400
I	Time, minutes	60	60	60	60	60	60	10	10	60	60	60	60	60	60	60	60
T	Coal Drying	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
I																	
O																	
N																	
S																	
	INPUT																
M	Solvent	247.3	219.1	245.5	234.2	238.6	229.4	239.8	240.5	237.2	232.8	235.0	241.0	237.4	235.3	239.4	232.0
A	Coal m.a.f	61.5	54.8	59.2	58.6	59.6	57.4	59.9	60.1	58.2	59.3	58.8	60.2	59.4	58.8	59.8	58.1
T	Ash	5.5	4.9	5.2	5.2	5.3	5.1	5.3	5.4	5.2	5.3	5.2	5.4	5.3	5.2	5.3	6.2
E	H_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	30.0	29.5	0.0
R	H_2	9.2	9.4	9.4	10.0	9.7	9.0	10.4	9.9	10.2	10.2	10.0	3.7	3.7	3.2	2.8	5.5
I	H_2S	5.6	8.6	11.4	0.0	0.0	8.8	0.0	8.5	0.0	0.0	9.0	8.2	0.0	0.0	8.8	9.0
A	CO	-	-	-	-	-	-	-	-	5.9	5.9	6.0	93.6	101.7	84.9	78.9	79.7
L	N_2	-	-	-	-	-	-	-	-	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
B	Catalyst	-	-	-	-	-	-	-	-	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
A	OUTPUT																
L	H_2O	4.5	6.2	5.0	9.0	6.2	5.8	4.1	3.3	2.4	1.2	3.6	0.0	0.0	20.4	11.5	0.0
A	Org. Lights	106.5	103.8	100.0	97.8	94.7	95.7	76.8	74.8	30.7	28.8	25.5	38.9	31.3	29.9	29.9	38.6
N	Heavy	92.8	93.4	115.7	95.4	129.0	122.3	186.4	190.7	225.9	203.2	237.8	224.1	226.9	227.5	240.7	227.3
C	Washings	27.9	10.8	8.5	3.9	6.1	5.8	17.9	9.2	21.4	48.1	22.3	18.7	21.9	20.9	16.3	11.7
E	Reactor solids	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	H_2	4.5	4.7	4.0	4.8	6.9	4.8	9.4	8.2	9.3	9.4	8.4	2.8	3.2	3.1	3.1	4.3
g.	CO_2	5.5	4.5	5.1	5.5	6.1	4.6	4.8	4.6	7.3	7.1	10.0	25.7	21.4	34.6	51.6	25.3
	CO	1.2	1.1	1.0	1.4	1.4	0.9	0.8	0.7	4.3	4.6	2.7	83.1	90.2	65.9	44.9	62.4
	H_2S	8.9	11.6	14.5	6.2	6.2	10.7	2.8	9.0	2.4	2.1	10.5	6.1	1.5	2.2	9.8	8.5
	C_1	13.9	12.5	14.6	14.7	14.2	10.9	3.0	3.1	1.4	1.3	1.4	1.7	1.6	1.4	1.4	1.5
	C_2	9.1	8.8	9.6	10.3	10.1	7.6	2.6	2.5	1.2	1.1	1.2	1.2	1.2	1.0	1.0	1.1
	C_3 C_4	11.5	12.3	12.9	15.2	13.9	10.2	6.3	6.5	4.3	3.8	2.1	8.6	5.2	3.8	3.6	7.4
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	87.0	90.9	88.0	85.7	94.1	90.1	99.8	99.3	99.5	97.8	100.4	99.7	99.2	98.3	98.9	99.4
R	IOR (g.)	38.8**	31.0**	27.6**	35.9**	63.5	41.4	37.0	28.6	16.3	15.3	18.8	31.3	38.7	22.1	14.5	27.7
E	Distillable (g.)	152.5	144.2	140.8	128.2	154.2	135.6	119.8	144.8	105.6	n.d.	118.7	120.5	116.0	97.4	102.3	107.7
S	C_1-C_4 (g.)	34.5	33.6	37.1	40.2	38.2	28.7	11.9	12.1	6.9	6.2	4.7	11.5	8.0	6.2	6.0	10.0
U	H_2 consumed (gr)	4.7)	(4.7)	(5.4)	(3.8)	(1.8)	(4.2)	(1.0)	(1.7)	(0.9)	(0.8)	(1.6)	(0.9)	(0.5)	(0.1)	0.3	(1.2)
L	H_2S (g.)	3.3	3.0	3.1	6.2	6.2	1.9	2.8	0.5	2.4	2.1	1.5	(2.1)	1.5	2.2	1.0	(0.5)
T	Hexane Insol. Liq. (g.)	41.0	33.4	37.9	43.2	8.0	34.7	60.9	62.0	86.5	80.8	59.4	58.4	60.0	66.9	78.9	62.1
S	S in hex ins.	1.5	1.2	1.4	1.4	0.3	n.d.	2.8	3.1	3.7	3.2	2.8	2.8	2.7	2.7	3.5	2.9
	H_2O (g.)	4.5	6.2	5.0	9.0	6.2	5.8	4.1	3.3	2.4	1.2	3.6	0.0	0.0	20.4	1.5	0.0
	Coversion, % m.a.f. coal	36.9**	43.4**	53.4**	38.7**	(6.5)**	27.9	38.2	52.4	72.1	74.2	68.0	48.0	47.3	62.4	75.9	52.2
	Distillate yield, % total m.a.f.	49.4	52.6	46.2	43.8	51.7	47.3	40.0	48.1	35.6	n.d.	40.4	40.0	39.1	33.1	34.2	36.4

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS CONDUCTED USING FORESTBURG COAL

	TEST #	69	69R	70	71	72	74	81	84	85	85R	91	92	93	95
R															
E															
A	Coal Type	F	F	F	F	F	F	F	F	F	F	F	F	F	F
C	Solvent	CLVB													
T	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	2/1	2/1	2/1	2/1	2/1	4/1
O	Gas Mixture	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2/H2S	H2	H2/H2S	H2/H2S	H2/H2S	H2	H2/H2S	H2/H2S	H2
R	H_2S %, m.a.f. coal	15	15	15	15	15	10	0	15	10	10	0	15	20	0
	CO/H_2, volume ratio	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C	H_2O, % m.a.f. coal	-	0.0	-	-	-	-	-	-	0.0	0.0	0.0	0.0	0.0	0.0
O	Catalyst, type	-	-	-	-	-	-	CoMo	CoMo	None	None	None	None	None	EMR
N	Pressure, MPa	17.2	17.2	17.2	17.2	13.8	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
D	Temperature °C	425	425	425	425	425	425	400	400	425	425	425	425	425	425
I	Time, minutes	60	60	60	90	60	60	60	60	60	60	60	60	60	60
T	Coal Drying	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
I															
O															
N															
S															
	INPUT														
M	Solvent	234.6	241.5	236.2	210.6	235.8	236.0	230.1	234.8	198.7	199.4	197.3	189.0	197.4	232.0
A	Coal m.a.f	58.6	60.4	59.0	52.7	58.9	59.0	57.5	58.7	99.4	99.7	98.7	94.5	98.7	58.0
T	Ash	5.3	6.5	5.3	4.7	5.3	5.2	5.2	5.2	8.9	8.9	8.8	8.5	8.8	8.0
E	H_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
R	H_2	9.6	10.4	9.8	10.2	7.4	10.1	11.4	11.0	10.4	10.5	10.9	10.0	9.6	10.8
I	H_2S	8.8	9.1	10.8	8.6	8.5	5.8	-	8.7	9.0	9.2	0.0	14.8	20.1	0.0
A	CO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
L	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
B	Catalyst	-	-	-	-	-	-	5.7	5.8	-	-	-	-	-	-
A	OUTPUT														
L	H_2O	5.0	4.6	5.8	4.7	5.1	4.8	5.3	8.0	9.2	8.8	7.6	11.0	11.7	8.9
A	Org. Lights	71.0	72.4	70.4	28.3	72.5	74.6	21.4	20.7	66.9	77.4	69.4	66.4	79.2	73.7
N	Heavy	173.4	198.2	184.8	192.7	174.5	174.4	245.3	250.9	190.2	152.0	180.7	183.7	180.7	186.7
C	Washings	16.6	8.4	10.0	15.3	17.3	18.2	14.1	12.2	12.6	43.0	16.1	9.0	6.5	11.6
E	Reactor solids	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	H_2	6.3	7.1	6.5	7.8	4.9	7.0	8.5	7.4	6.0	6.6	8.2	8.3	5.7	6.8
G.	CO_2	4.5	4.6	4.7	3.9	4.8	4.6	2.8	2.6	7.5	8.6	9.0	8.2	8.0	4.3
	CO	0.7	0.7	0.7	0.6	0.6	0.9	1.3	0.9	1.0	0.8	1.1	0.8	0.6	1.0
	H_2S	10.1	10.9	12.1	10.6	9.6	8.5	3.5	12.9	11.0	10.4	3.0	14.4	18.0	4.3
	C_1	4.3	4.0	4.4	1.8	4.4	4.5	1.1	1.2	4.5	4.6	4.0	4.7	5.4	3.3
	C_2	3.3	3.1	3.4	1.5	3.3	3.5	1.0	1.1	3.4	3.4	3.2	3.5	3.9	2.5
	C_3 C_4	6.6	6.8	6.7	4.4	5.9	7.2	3.2	3.9	6.4	6.4	6.3	6.6	6.9	5.0
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	95.2	97.7	96.4	94.7	96.0	97.5	99.2	99.3	97.6	98.3	97.6	99.3	97.6	99.9
R	IOR (g.)	18.3	11.7	17.9	15.1	21.8	16.8	26.1	21.8	14.6	17.6	39.6	17.3	20.0	4.1
E	Distillable (g.)	169.5	171.6	175.3	104.6	167.3	170.0	91.3	73.3	139.2	n.d.	126.7	141.7	149.6	154.0
S	C_1-C_4 (g.)	14.2	13.9	14.5	7.7	13.6	15.2	5.3	6.2	14.3	14.4	13.5	14.8	16.2	10.8
U	H_2 consumed (gr)	(3.3)	(3.3)	(3.3)	(2.4)	(2.5)	(3.1)	(2.9)	(3.6)	(4.6)	(3.9)	(2.7)	(3.7)	(3.9)	(4.0)
L	H_2S (g.)	1.3	1.8	1.3	2.0	1.1	2.7	3.5	4.2	2.0	1.2	3.0	(0.4)	(2.1)	4.3
T	Hexane Insol. Liq. (g.)	60.4	63.5	59.4	50.0	59.9	62.0	51.6	100.5	80.2	72.5	70.4	75.73	74.6	54.1
S	S in hex ins.	2.6	n.d.	2.6	2.2	2.7	2.7	1.7	3.5	3.2	n.d.	2.3	2.9	2.8	3.2
	H_2O (g.)	5.0	4.6	5.8	4.7	5.1	4.8	5.3	8.0	9.2	8.8	7.6	11.0	11.7	8.9
	Coversion, % m.a.f. coal	68.7	80.7	69.7	71.3	63.0	71.5	54.6	62.9	85.3	82.3	59.8	81.7	79.7	92.9
	Distillate yield, % total m.a.f.	57.8	56.8	59.4	39.7	56.8	57.6	31.8	25.0	46.7	n.d.	42.7	50.1	50.1	53.1

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS USING 1 METHYL NAPTHALENE AS SOLVENT

	TEST #	7	8	9	13	14	15	25	26	27	33	38	39	43	44	45	75	76
REACTION CONDITIONS	Coal Type	F	F	F	F	F	F	F	L	L	F	L	L	L	L	F	F	F
	Solvent	MN																
	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
	Gas Mixture	N2	H2/H2S	H2/H2S	N2	N2/H2S	N2/H2S	H2/H2S	H2/H2S	H2	H2	H2/CO/H2S	H2/CO	H2/CO/H2S	H2/CO	N2/H2S	H2/H2S	
	H_2S %, m.a.f. coal	0	10	10	0	10	10	10	10	0	0	10	0	10	0	10	15	
	CO/H_2, volume ratio	-	-	-	-	-	-	-	-	-	-	2/1	2/1	2/1	2/1	-	-	
	H_2O, % m.a.f. coal	27	27	27	27	27	27	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0	0	0
	Catalyst, type	None																
	Pressure, MPa	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25
	Temperature °C	425	425	425	425	425	425	425	425	425	425	425	425	425	425	425	425	425
	Time, minutes	30	60	30	30	60	30	30	60	60	60	120	120	60	60	60	60	60
	Coal Drying	WET	WET	WET	WET	WET	WET	OD	OD	OD	OD	OD	OD	OD	OD	OD	SD	SD
MATERIAL BALANCE	INPUT																	
	Solvent	222.8	218.2	216.4	225.7	221.6	224.2	243.2	240.4	229.5	245.3	221.1	228.8	231.6	233.6	237.7	232.8	253.0
	Coal m.a.f	54.4	54.3	53.9	56.5	55.5	56.1	60.8	60.0	57.3	61.3	55.2	57.1	57.7	58.4	53.7	58.2	64.8
	Ash	4.9	5.0	5.0	4.8	4.7	4.8	5.4	12.2	11.6	5.5	11.2	11.6	11.8	12.0	4.8	5.2	5.7
	H_2O	14.9	14.7	14.5	15.1	14.8	15.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	H_2	9.8	8.8	8.8	-	-	-	10.2	9.4	9.8	10.4	3.3	3.4	3.4	3.4	-	10.0	10.8
	H_2S	-	5.6	5.6	-	5.7	5.7	5.5	5.5	0.0	0.0	5.5	0.0	5.5	0.0	5.4	8.5	0.0
	CO	-	-	-	-	-	-	-	-	-	0.0	93.8	97.2	97.2	97.2	-	0.0	0.0
	N_2	-	-	-	123.8	104.2	119.0	-	-	-	0.0	0.0	0.0	0.0	0.0	143.2	0.0	0.0
	Catalyst	None																
	OUTPUT																	
	H_2O	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.7
	Org. Lights	79.3	109.0	102.5	133.2	99.2	124.9	106.6	103.0	110.6	142.6	139.0	140.2	154.8	139.1	166.6	131.7	135.7
	Heavy	160.8	132.7	132.0	105.4	173.0	132.1	184.6	190.8	169.6	160.7	127.6	139.6	131.1	120.2	86.7	143.7	168.2
	Washings	38.0	35.7	39.3	46.0	-	18.5	-	-	-	0.0	-	-	13.0	36.7	43.0	5.5	3.6
	Reactor solids	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	H_2	8.9	7.7	7.2	0.0	0.0	0.0	8.6	7.2	8.0	8.2	2.0	2.4	2.7	2.9	0.0	8.3	9.8
g.	CO_2	4.6	7.0	6.6	5.3	6.3	4.8	4.2	5.0	4.6	4.3	24.1	21.8	28.7	24.1	6.8	3.5	4.9
	CO	0.4	1.7	1.6	0.4	0.0	0.0	0.5	1.5	0.7	1.0	85.4	83.9	72.4	74.8	0.0	0.8	0.7
	H_2S	0.0	8.1	7.5	0.0	4.1	3.3	4.7	5.0	0.1	0.0	1.9	0.0	2.7	-	3.9	5.5	0.1
	C_1	0.8	1.2	1.2	0.4	0.8	0.4	1.4	3.8	2.0	2.5	2.6	2.3	2.6	1.9	0.8	3.1	2.0
	C_2	0.3	0.7	0.6	0.2	0.3	0.2	0.4	0.8	0.4	0.9	0.7	0.6	0.7	0.5	0.3	0.7	0.5
	C_3 C_4	1.6	3.4	3.2	2.6	2.0	2.6	1.8	2.1	1.0	2.1	4.0	0.2	3.6	1.8	1.8	1.6	-
	N_2	-	-	-	124.6	107.3	123.3	-	-	-	0.0	0.0	0.0	0.0	0.0	144.6	0.0	0.0
	Mat. Bal. %	95.2	100.1	99.2	98.1	96.6	96.5	96.2	97.5	96.4	100.0	99.3	98.3	101.2	99.4	100.7	96.8	97.6
RESULTS	IOR (g.)	29.6	24.5	29.2	33.0	48.9	40.0	24.1	12.4	14.3	26.7	17.1	23.3	18.3	19.1	56.8	11.1	25.4
	Distillable (g.)	n.d.	n.d.	n.d.	n.d	n.d.	n.d.	n.d.	n.d.	n.d.	n.d	228.3	n.d.	n.d.	n.d.	241.2	n.d.	n.d.
	C_1-C_4 (g.)	2.7	5.3	5.0	2.8	3.1	3.2	3.6	6.7	3.4	5.5	7.3	3.1	6.9	4.2	2.9	5.4	2.5
	H_2 consumed (gr)	(0.9)	(1.1)	(1.6)	-	-	-	(1.6)	(2.2)	(1.8)	(2.2)	(1.3)	(1.2)	(0.7)	(0.5)	-	(1.7)	(1.0)
	H_2S (g.)	0.0	2.5	1.9	0.0	(1.6)	(2.4)	(0.8)	(0.5)	0.0	0.0	(3.6)	(0.0)	(2.8)	0.0	(1.5)	(3.0)	0.0
	Hexane Insol. Liq. (g.)	10.4	18.3	9.4	6.1	n.d.	8.2	18.5	23.5	21.0	23.7	9.2	11.5	15.0	15.7	0.0	19.7	30.6
	S in hex ins.	0.6	1.5	1.3	0.0	n.d.	0.1	0.7	0.7	0.5	1.8	0.8	0.5	0.3	0.1	0.0	0.4	0.0
	H_2O (g.)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d	n.d	n.d	n.d	n.d	n.d.	n.d.	n.d.	n.d.	n.d.
	Coversion, % m.a.f. coal	45.5	54.9	45.8	41.6	11.9	28.7	60.4	79.3	75.0	56.4	69.0	59.2	68.3	67.3	(5.7)	80.9	60.8
	Distillate yield, % total m.a.f.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	82.6	n.d.	n.d.	n.d.	82.6	n.d.	n.d.

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

TESTS CONDUCTED USING LIGNITE AND CLVB

	TEST #	29	30	36	37	40	79	80	42
R									
E									
A	Coal Type	L →							
C	Solvent	CLVB →							
T	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
O	Gas Mixture	H2/H2S	H2	H2/CO	H2/CO/H2S	H2/CO/H2S	H2/CO	H2/CO/H2S	H2/CO/H2S
R	H_2S %, m.a.f. coal	10	0	0	10	10	0	15	10
	CO/H_2, volume ratio	-	-	2:1	2:1	2:1	1:1	1:1	2:1
C	H_2O, % m.a.f. coal	0.0	0.0	0.0	0.0	0.0	50.0	50.0	0.0
O	Catalyst, type	None →							
N	Pressure, MPa	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
D	Temperature °C	425	425	425	425	425	400	400	425
I	Time, minutes	60	60	120	120	60	60	60	120
T	Coal Drying	OD	OD	OD	OD	OD	SD	SD	OD
I									
O									
M									
S									
	INPUT								
M	Solvent	223.2	236.6	225.7	220.2	223.0	250.5	242.5	226.3
A	Coal m.a.f	55.7	59.1	56.4	55.0	55.7	62.6	60.6	56.5
T	Ash	11.3	12.0	11.4	11.2	11.3	12.8	12.5	11.5
E	H_2O	0.0	0.0	0.0	0.0	0.0	28.8	28.8	0.0
R	H_2	10.0	10.4	3.3	3.3	3.3	3.9	4.4	3.3
I	H_2S	5.7	0.0	0.0	5.5	5.5	0.0	8.7	5.5
A	CO	0.0	0.0	94.7	94.7	94.7	56.7	58.7	94.7
L	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
S	Catalyst	-	-	-	-	-	-		
A	OUTPUT								
L	H_2O	n.d.	n.d.	n.d.	n.d.	n.d.	17.4	8.6	n.d
A	Org. Lights	73.6	69.8	96.3	96.1	81.5	30.9	25.7	97.4
N	Heavy	170.2	175.4	82.4	21.8	162.8	254.0	242.1	12.5
C	Washings	19.9	24.8	63.6	59.5	16.0	20.4	26.1	77.5
E	Reactor solids	-	-	-	102.1	-	-	-	62.3
	H_2	6.1	6.8	1.3	0.8	1.7	3.4	4.3	1.0
g.	CO_2	5.2	5.6	31.4	32.3	26.4	38.5	58.0	32.1
	CO	2.3	1.9	66.2	77.1	77.2	34.1	29.8	76.7
	H_2S	9.2	5.5	3.6	3.7	4.3	2.5	9.7	4.3
	C_1	4.2	4.7	7.6	7.7	3.8	1.3	1.2	6.4
	C_2	3.3	3.6	5.6	5.3	3.0	1.1	1.0	4.8
	C_3 C_4	6.0	6.2	11.4	11.5	12.8	3.7	3.7	14.4
	N_2								
	Mat. Bal. %	98.1	96.0	94.3	107.2	99.0	98.1	98.6	98.1
R	IOR (g.)	14.6	16.9	35.4	113.2	35.7	25.3	16.4	60.7
E	Distillable (g.)	162.2	165.5	146.6	130.6	149.3	114.8	107.6	n.d.
S	C_1-C_4 (g.)	13.5	14.5	24.6	24.4	19.6	6.1	5.9	25.6
U	H_2 consummed (gr)	(3.9)	(3.6)	(2.0)	(2.5)	(1.6)	(0.5)	(0.1)	(2.3)
L	H_2S (g.)	3.7	5.5	0.0	(1.8)	(1.2)	0.0	1.0	(1.2)
T	Hexane Insol. Liq. (g.)	55.8	56.8	34.1	11.7	40.9	68.9	66.5	28.6
S	S in hex ins.	3.3	2.0	3.6	0.5	1.2	2.8	2.8	1.3
	H_2O (g.)	n.d.	n.d.	n.d.	n.d	n.d	17.4	8.6	n.d.
	Coversion, % m.a.f. coal	73.8	71.4	37.2	(105.8)	35.9	59.4	73.0	(7.4)
	Distillate yield, % total m.a.f.	56.3	56.0	52.0	47.5	53.6	36.6	32.5	n.d.

Sandwell Beak Research Group
Energy and Environmental Science

TESTING AND DEMONSTRATION OF THE USE AND EFFECTIVENESS OF HYDROGEN SULPHIDE AS A CATALYST IN THE CO-PROCESSING OF ESTEVAN LIGNITES, FORESTBURG SUB-BITUNIMOUS COAL AND COLD LAKE VACUUM RESIDUE

FINAL REPORT

BY

SANDWELL BEAK RESEARCH GROUP

Work on this project was conducted under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources, Canada

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CANMET SCIENTIFIC AUTHORITY: Mr. P.M. Rahimi

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A PROMOTER IN COAL LIQUEFACTION

September 1983

	TEST #	96	97	98	99	100R	101	102	103	104	105	101R	102R	103R	106
REACTION CONDITIONS	Coal Type	F CLUB	F CLUR	F CLUR	F CLUB	F CLUB	F CLUB	F CLUB	F CLUB	F CLUR	F CLUB	F CLUR	F CLUR	F CLUB	F CLUB
	Solvent	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1
	S/C, wt ratio	H_2	H_2	H_2/CO/HS	H_2/CO/HS	H_2/CO	H_2/CO/HS	H_2/CO/HS	H_2/CO/HS	H_2/	HS	H_2/ HS	H_2/CO/HS	H_2/CO/HS	H_2
	Gas Mixture	0	0	20	10	0	10	15	20	15	15	10	15	20	0
	H_2S %, m.a.f. coal	-	-	1:1	1:1	1:1	1:1	1:1	1:1	-	-	1:1	1:1	1:1	-
	CO/H_2, volume ratio	0	0	50	50	50	50	50	50	0	0	50	50	50	0
	H_2O, % m.a.f. coal	EMR	-	-	-	-	-	-	-	-	-	-	-	-	-
	Catalyst, type	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25	17.25
	Pressure, MPa	400	425	400	400	425	425	425	425	375	450	425	425	425	450
	Temperature °C	60	60	60	60	60	60	60	60	60	20	60	60	60	20
	Time, minutes	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
	Coal Drying														
MASS BALANCE	INPUT	235.5	223.3	218.3	239.8	238.2	238.5	232.5	238.0	232.0	238.5	231.5	234.0	229.9	231.6
	Solvent	58.9	55.8	54.6	60.0	59.6	59.7	57.5	59.2	58.0	59.6	57.9	58.5	57.5	58.4
	Coal m.a.f.	8.1	5.8	5.6	6.2	6.1	6.1	6.0	6.1	6.0	6.1	6.0	6.0	5.8	6.0
	Ash	0.0	0.0	25.0	29.3	29.4	29.3	29.4	29.4	0.0	0.0	29.4	29.4	29.4	0.0
	H_2O	11.3	10.4	4.8	4.1	4.0	3.5	4.0	3.8	11.9	9.9	3.8	4.0	3.8	17.3
	H_2	0.0	0.0	9.9	6.0	0.0	5.6	8.4	12.2	9.7	9.5	6.0	8.4	12.2	0.0
	H_2S	0.0	0.0	61.7	60.3	57.1	53.7	57.3	49.0	0.0	0.0	54.3	51.3	49.0	0.0
	CO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Catalyst														
	OUTPUT	7.2	7.5	9.7	27.0	17.6	10.7	9.0	10.6	6.8	6.0	11.7	10.1	9.5	5.8
	H_2O	19.2	76.8	29.3	21.8	94.3	93.8	97.1	75.3	9.3	94.0	86.4	77.6	86.4	93.5
	Org. Lights	243.5	158.0	204.1	226.8	137.2	178.7	157.5	184.4	252.7	146.7	156.1	166.3	162.8	142.3
	Heavy	23.0	17.3	32.2	28.6	46.4	23.1	8.0	12.1	23.0	23.0	20.8	15.6	18.1	27.3
	Washings	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Reactor solids	8.4	8.4	4.6	4.0	3.8	3.7	3.9	3.5	10.7	7.4	3.9	3.7	3.7	8.3
	H_2	3.4	4.7	49.8	42.2	40.3	55.5	63.0	61.0	3.6	4.9	55.4	62.0	64.2	5.1
	CO_2	0.4	0.7	31.1	33.6	35.4	25.1	15.0	16.9	0.0	0.7	20.8	18.6	15.9	0.8
	CO	1.8	3.2	10.8	6.8	3.4	7.2	10.8	12.7	9.3	9.3	7.2	9.7	12.1	3.4
	H_2S	0.9	4.0	1.4	0.3	4.9	4.7	5.1	4.6	0.3	5.3	4.7	5.0	5.0	5.1
	C_1	0.7	3.1	1.1	1.0	3.7	3.5	3.6	3.3	0.3	4.0	3.4	3.5	3.5	4.2
	C_2	2.9	5.8	4.7	4.9	7.5	7.5	8.4	7.2	1.7	6.9	7.1	6.7	6.6	7.1
	C_3 C_4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	N_2	99.2	98.2	99.7	97.8	100.1	104.3	98.0	98.1	100.0	96.2	97.2	96.7	100.0	98.2
	Mat. Bal. %	8.3	17.5	13.3	13.2	32.1	19.1	14.7	14.9	37.3	26.6	18.7	15.3	15.8	37.3
RESULTS	IOR (g.)	86.3	159.1	106.5	110.9	157.0	170.3	154.9	175.6	74.4	155.6	nd	nd	nd	147.5
	Distillable (g.)	4.5	12.9	7.2	6.2	16.1	15.7	17.1	15.1	2.3	16.2	15.2	15.2	15.1	16.4
	C_1-C_4 (g.)	(2.9)	(2.0)	(0.2)	(0.1)	(0.2)	(0.2)	(0.1)	(0.3)	(1.2)	(2.5)	0.1	(0.3)	(0.1)	(2.7)
	H_2 consumed (gr)	1.8	3.2	0.9	0.8	3.4	1.6	2.4	0.5	(0.4)	(0.6)	1.2	1.3	(0.1)	3.4
	H_2S (g.)	79.3	66.5	69.9	73.5	61.3	63.0	62.8	nd	64.4	66.3	nd	nd	70.9	60.7
	Hexane Insol. Liq. (g.)		2.79	3.14	3.27	2.76	2.76	2.35	2.63	2.83	2.98	2.85	1.8	3.17	2.59
	S in hex ins.	7.2	7.5	(15.3)	(2.3)	(11.8)	(18.6)	(20.4)	(18.8)	6.8	6.0	(17.7)	(19.3)	(19.9)	5.8
	H_2O (g.)														
	Conversion, % m.a.f. coal	85.8	68.6	75.6	78.0	46.1	67.8	74.4	74.8	35.7	55.3	67.7	73.9	72.5	35.0
	Distillate yield, % total m.a.f.	29.3	57.0	39.0	37.0	52.7	57.1	53.4	59.0	25.6	52.2	nd	nd	nd	50.7

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A CATALYST IN THE COPROCESSING OF ESTEVAN LIGNITES, FORESTBURG SUB-BITUMINOUS COALS WITH COLD LAKE VACUUM RESIDUE

	TEST #	107	108	109	110	111	112	113	108R	107R	115	116	117	118
REACTOR CONDITIONS	Coal Type	L	L	L	L	L	L	L	L Vacuum evacuated	L	L	L	None	None
	Solvent	CLVB	CLVB	CLVB	CLVB	CLVB	CLVB	CLVb	CLVB	CLVB	HAO	HAO	CLVB	CLVB
	S/C, wt ratio	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	4/1	--	--
	Gas Mixture	H2	H2/CO	H2/CO/H2S	H2/CO/H2S	H2/CO/H2S	H2/CO/H2S	H2/CO/H2S	CO/H2	H2	H2/CO	H2/CO/H2S	H2/CO/H2S	H2/CO
	H_2S %, m.a.f. coal	--	--	10	15	10	10	10	50	--	--	10	10	10
	CO/H_2, volume ratio	--	1.1	1.1	1/1	1/1	1/1	1/1	1/1	--	1/1	1/1	1/1	1/1
	H_2O, % m.a.f. coal	--	50	50	50	50	50	50	50	--	50	50	50	50
	Catalyst, type	--	--	--	--	--	--	--	--	--	--	50	--	--
	Pressure, MPa	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.2
	Temperature °C	425	425	425	425	425	425	425	425	425	425	425	425	425
	Time, minutes	60	60	60	60	10	20	40	60	60	60	60	60	60
	Coal Drying	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	--	--
MATERIAL BALANCE g.	INPUT													
	Solvent	236.5	235.0	239.5	232.0	233.7	234.1	231.8	234.3	227.6	258.3	232.8	279.5	292.2
	Coal m.a.f	54.4	58.8	59.9	58.2	58.4	58.5	58.0	58.6	56.9	64.6	58.2	0.0	0.0
	Ash	11.2	12.0	12.3	11.9	12.0	12.0	11.9	12.0	11.7	13.2	11.9	0.0	0.0
	H_2O	0.0	28.8	28.8	28.8	28.8	28.8	28.8	28.8	0.0	30.2	28.9	29.0	28.8
	H_2	10.8	4.3	4.1	3.7	4.1	4.0	4.0	4.3	11.4	4.3	4.0	4.1	4.3
	H_2S	0.0	0.0	6.0	8.0	6.0	6.1	5.9	0.0	0.0	0.0	6.1	5.9	0.0
	CO	0.0	55.5	53.2	48.8	53.5	56.7	54.5	56.7	0.0	55.7	58.0	54.9	55.8
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Catalyst	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	OUTPUT													
	H_2O	6.4	14.5	8.0	8.1	17.1	12.4	9.8	13.9	6.0	4.9	17.0	8.8	15.1
	Org. Lights	61.9	63.1	61.8	63.9	26.3	34.7	50.8	63.2	55.1	66.0	42.2	68.9	72.9
	Heavy	195.4	195.8	201.0	192.2	244.6	225.6	211.4	192.7	200.8	255.9	238.8	180.9	177.6
	Washings	12.8	17.0	22.9	20.1	18.4	30.1	19.2	17.4	15.5	10.8	7.5	15.7	16.9
	Reactor solids	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	H_2	7.5	3.9	3.7	3.6	4.4	4.1	3.9	4.0	8.5	4.7	4.6	4.7	4.5
	CO_2	4.7	47.1	60.3	62.2	36.9	44.8	56.2	49.4	5.0	35.0	45.7	33.8	16.8
	CO	1.1	26.1	18.3	16.3	34.6	29.2	23.2	28.9	1.3	38.5	30.4	34.0	45.9
	H_2S	3.8	3.4	7.7	10.1	6.5	7.2	7.6	3.5	3.5	0.2	4.9	8.2	3.4
	C_1	3.6	3.5	3.3	3.6	1.0	1.3	2.4	3.3	2.8	1.0	0.9	3.1	3.0
	C_2	2.7	2.7	2.4	2.5	0.7	1.1	1.9	2.7	2.2	0.9	0.8	2.6	2.8
	C_3 C_4	5.4	6.3	5.0	5.7	1.5	4.2	4.8	5.9	4.9	1.8	1.7	6.3	6.1
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	97.6	97.4	97.9	98.9	98.8	98.6	99.2	97.5	99.4	98.4	98.7	98.4	95.8
RESULTS	IOR (g.)	15.7	22.7	14.6	15.7	30.9	25.1	17.7	21.1	19.1	7.6	7.9	6.7	8.4
	Distillable (g.)	158.2	131.4	158.6	156.9	95.7	120.4	141.3	143.6	144.2	269.7	227.6	119.8	159.4
	C_1-C_4 (g.)	11.7	12.5	11.6	11.8	3.2	6.6	9.1	11.9	9.9	3.7	3.4	12.0	11.9
	H_2 consumed (gr)	(3.3)	(0.5)	(0.4)	(0.1)	0.3	0.1	(0.1)	(0.3)	(2.9)	0.4	0.6	0.6	0.2
	H_2S (g.)	3.8	3.4	1.7	2.1	0.5	1.1	1.7	3.5	3.5	0.2	4.9	2.3	3.4
	Hexane Insol. Liq. (g.)	60.0	62.6	63.6	57.3	61.8	63.2	60.1	59.8	58.5	43.8	36.2	55.6	62.2
	S in hex ins.	2.4	2.7	2.9	2.5	2.78	2.9	2.7	2.6	2.2	0.4	0.5	4.0	4.6
	H_2O (g.)	6.4	(14.5)	(20.8)	(20.7)	(11.7)	(16.4)	(19.0)	(14.8)	6.0	(25.3)	(11.9)	(20.2)	(13.7)
	Coversion, % m.a.f. coal	71.1	61.4	75.6	73.0	47.2	57.1	69.5	64.0	66.5	88.2	86.4		
	Distillate yield, % total m.a.f	54.4	44.7	53.0	53.9	32.7	41.1	48.8	50.1	51.1	17.8	?	42,9	54.5

Sandwell Bank Research Group
Energy and Environmental Science

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A CATALYST IN THE COPROCESSING OF ESTEVAN LIGNITES, FORESTBURG SUB-BITUMINOUS COALS WITH COLD LAKE VACUUM RESIDUE

	TEST #	59R2	55R2	55R3	23R1	69R	10R	114
R							Vacuum evacuated	
E								
A	Coal Type	F	F	F	F	F	F	F
C	Solvent	CLVB Old	CLVB	CLVB Old	CLVB Old	CLVB	CLVB	Tetralin
T	S/C, wt ratio	411	4/1	4/1	4/1	4/1	4/1	4/1
O	Gas Mixture	H2	H2/H2S	H2H2S	H2	H2H2S	H2	H2
R	H_2S %, m.a.f. coal	--	15	15	--	15	--	--
	CO/H_2, volume ratio	--	--	--	--	--	--	--
C	H_2O, % m.a.f. coal	--	--	--	--	--	--	--
O	Catalyst, type	--	--	--	--	--	--	--
N	Pressure, MPa	--	--	--	--	--	--	--
D	Temperature °C	17.2	17.2	17.2	17.2	17.2	17.2	17.2
I	Time, minutes	375	400	400	400	425	425	425
T	Coal Drying	60	60	60	60	60	60	60
I		SD	SD	SD	SD	SD	SD	SD
O								
N								
S								
	INPUT							
M	Solvent	236.7	239.1	236.3	233.5	229.7	233.2	228.4
A	Coal m.a.f	59.2	59.8	59.1	58.4	57.4	58.3	54.6
T	Ash	6.3	6.2	6.2	6.0	5.9	6.0	5.7
E	H_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0
R	H_2	12.0	10.8	10.8	11.3	10.2	10.6	11.2
I	H_2S	0.0	9.2	9.2	0.0.	9.2	0.0	0.0
A	CO	0.0	0.0	0.0	0.0	0.0	0.0	0.0
L	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
B	Catalyst	0.0	0.0	0.0	0.0	0.0	0.0	0.0
A	OUTPUT							
L	H_2O	4.7	6.4	7.3	4.5	6.0	4.9	n.d.
A	Org. Lights	14.4	34.3	25.3	31.8	78.5	70.5	100.1
N	Heavy	245.7	234.9	243.6	230.8	175.2	180.6	168.9
C	Washings	30.3	20.9	12.1	13.6	10.8	18.1	13.1
E	Reactor solids	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	H_2	11.2	8.9	9.0	9.9	7.0	8.8	10.7
g.	CO_2	3.9	4.1	3.9	4.6	4.3	4.9	4.0
	CO	0.4	0.4	0.4	0.4	0.6	0.5	0.7
	H_2S	1.2	9.6	9.5	2.2	10.6	3.6	0.4
	C_1	0.5	1.1	1.1	1.2	4.3	3.6	0.8
	C_2	0.4	1.0	1.0	1.1	3.3	3.1	0.4
	C_3 C_4	2.9	3.3	3.4	4.2	6.4	6.8	0.0
	N_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Mat. Bal. %	100.4	99.9	98.4	98.4	98.2	99.1	99.6
R	IOR (g.)	44.7	24.2	22.9	35.0	13.8	22.8	5.2
E	Distillable (g.)	84.1	122.4	101.8	104.9	157.3	144.6	n.d.
S	C_1-C_4 (g.)	3.8	5.4	5.5	6.5	14.0	13.5	1.2
U	H_2 consumed (gr)	(0.8)	(1.9)	(1.8)	(1.4)	(3.2)	(1.8)	(0.5)
L	H_2S (g.)	1.2	0.4	0.3	2.2	1.4	3.6	0.4
T	Hexane Insol. Liq. (g.)	67.1	69.1	73.1	68.4	69.0	63.8	22.3
S	S in hex ins.	2.5	3.0	3.1	2.7	3.0	2.68	0.1
	H_2O (g.)	4.7	6.4	7.3	4.5	6.0	4.9	n.d.
	Coversion, % m.a.f. coal	25.0	59.5	61.2	40.0	76.0	60.9	90.4
	Distillate yield, % total m.a.f.	28.9	41.0	34.5	35.9	54.8	49.6	n.d.

Sandwell Beak Research Group
Energy and Environmental Science

SUMMARY OF RESULTS, DEMONSTRATION AND TESTING OF THE USE OF HYDROGEN SULPHIDE AS A CATALYST IN THE COPROCESSING OF ESTEVAN LIGNITES, FORESTBURG SUB-BITUMINOUS COALS WITH COLD LAKE VACUUM RESIDUE

Notes:

nd = not determined
na = not applicable
Coal types: F = Forestburg subbituminous
L = Estevan lignite
Solvent types: CLVB = Cold Lake Vacuum Bottoms
MN = 1 mehtyl naphthalene
HAO = hydrogenated anthracene oil
AO = raw anthracene oil

S/C = weight ratio of solvent to m.a.f. coal
H2S% = calculated as % wt of m.a.f. coal or its equivalent
CO/H2 = volumen ratio of gases
H2O% = calculated at % wt of m.a.f. coal
coal drying = SD - slurry dried
OD - oven dried
Wet - not dried

OUTPUT: H2O - water physically separated from organic lights
Org lights - fraction condensed off vapour phase vented after reaction complete
Heavy - liquid / solid material collected in the cold receiver situated right under reactor
Washings - reactor product recovered from reactor and cold receiver by washing with THF, weight after THF removal
Reactor Solids - solid material recovered by mechanical means from reactor walls

RESULTS: IOR - THF insoluble organic matter calculated as total THF insoluble less ash
Hexane insoluble liquid total hexane insolubles less THF insolubles. (asphaltenes / preasphaltenes)

$$\text{conversion:} = \frac{\text{m.a.f. coal} - \text{IOR}}{\text{m.a.f. coal}} \times 100$$

$$\text{distillate yield} = \frac{\text{Distillate}}{\text{m.a.f coal + solvent}} \times 100$$

"Vacuum evacuated" indicates that in preparation for this test, the reactor was maintained under vacuum of 1mmHg or less and 450 degree C for 2 hours

DEMONSTRATION OF THE EFFECTIVENESS OF THE USE OF H2S AS A CATALYST IN THE CO-PROCESSING OF COALS WITH COLD LAKE VACCUM RESIDUE

TESTING PROCEDURES

The feed gases are introduced into the empty reactor at predetermined cold partial pressures measured on the pressure gauges installed for this purpose. When water is used, the preweighed amount is added to the empty reactor (before introducing the gases). The air is completely purged from the unit with nitrogen followed by hydrogen before the introduction of feed gases. The total volume of the feed gases is measured by way of a "blank run" followed by venting through a wet-test gas meter. Gas samples are taken under stirring and the composition is analysed on a Carle gas chromatograph. The partial pressures of the gases are adjusted, if necessary, until the required composition is obtained on the gas chromatograph. The reactor is then heated to the reaction temperature.

The preweighed solvent and coal (as received) are mixed in an open container at room temperature to obtain the feed slurry. The feed slurry is gradually heated to about 105-110 degree C on a hot plate. It is kept at this temperature under continual stirring until all water in the coal is completely evaporated. The completeness of water removal is checked by weighing the container repeatedly until constant weight is obtained (or the calculated amount of water, corresponding to the coal moisture analysis is removed). When Cold Lake Vacuum Bottoms is used as the solvent, the dried feed slurry is further heated to between 130-140 degree C before it is drawn into the hydraulic cylinder to ensure fluidity. The hydraulic cylinder is heated to 90 degree C. The required amount of feed slurry is drawn into the hydraulic cylinder by moving the piston. The reactor mixer is switched on.

The slurry is injected instantaneously into the reactor. The exact weight of slurry injected into the reactor is obtained by the weight difference of the feed slurry container before and after injection. The small amount of slurry which remains in the feed line is collected, weighed and taken into account.

After the injection of the feed slurry, the temperature of the reactor returns to the reaction temperature within 2-4 minutes. The temperature of the reacting slurry is continually recorded. Readings of the reactor temperature and pressure are taken at regular intervals throughout the test.

At the end of reaction time, the heater of the reactor is switched off. The slurry in the reactor is discharged into a 300 ml heavy receiver (316SS, high pressure, provided with bottom valve, top screw closure and electric heater). The top valve of the reactor is opened to release the hot gas phase. The gas phase containing unreacted gases, light fraction, water and hydrocarbon gases is passed through a water condenser followed by an ice/salt trap and the wet-test gas meter before being vented into a agueous NaOH solution scrubber. Upstream of the wet-test meter there is a pressure regulator and a micrometering valve which are used for flow control of the gases.

The light hydrocarbon and water vapours which condense in the water condenser are collected in a 300 ml vessel, designated the lights receiver. The vapours which condense in the ice-salt trap are added to the liquid collected in the lights receiver. The gas is repeatedly (at least twice) sampled during the venting operation downstream the ice/salt trap. At this point the pressure is less than 100 psi. The gas is analysed on the gas chromatograph.

After the collection of the light liquid fraction and the venting of gases, the heavy receiver is disconnected from the unit. The vessel is opened and the heavy material is collected by mechanical means. The reactor and heavy receiver are then washed with THF to recover the heavy material which remained in the reactor on the walls and stirrer. This material is designated as "washings", and it is recovered from THF in a vacuum rotary evaporator. The washings are weighed and added to the material collected in the heavy receiver. The water present in the light fraction is separated visually and both the water fraction and the organic lights are weighed to the closest .1g. The material balance is calculated for each test and only plus 1 to minus 6% is considered adequate. Test having a "poorer" material balance are repeated. A representative sample of the heavy and washings is used for determination of the THF insolubles and the hexane insolubles. The hexane insolubles precipitate is analysed for sulphur content using a Leco Analyser. A prorated representative composite sample of organic lights, heavy and washings is subjected to vacuum distillation - the analytical procedures are attached.

DETERMINATION OF HEXANE INSOLUBLES IN COAL-DERIVED LIQUIDS

Weigh to the nearest miligram a 400ml beaker containing a stirring rod. Add 10gr of thoroughly mixed liquid product and weigh to the nearest miligram. Add a small amount (maximum 5ml) of benzene and warm the beaker on a hot bath while mixing well. When the material is fluid and well mixed, add rapidly 250ml of hexane stirring vigorously. If sample is liquid enough, 20 volumes of hexane can be added to 1 volume of sample, without adding benzene at all.

Maintain the beaker under stirring at room temperature overnight. Place a tared fluted filter paper (previously dried at 105 degree C) into a large funnel and place a 600ml beaker under the funnel. Transfer the britle, finely divided insoluble material to the filter paper, continue washing with hexane until the washings are a pale yellow colour. Transfer the paper and the precipitated asphaltenes to the cleaned 250ml beaker with stirring rod and dry in a vacuum oven at 60 degree C for one hour. Cool in the dessicator for 20 minutes and weigh quickly.

Calculate the hexane insolubles by dividing the weight of the insoluble material in grams by the sample weight in grams and multiply. by 100. Subtract the THF insoluble percent in order to obtain the asphltene - preasphaltene fraction in percentage weight.

DETERMINATION OF THE THF INSOLUBLE

Weigh to the nearest 0.1mg a 100 ml beaker. Pour about 1-3gr of the thoroughly mixed sample in the beaker and weigh to the nearest 0.1mg. (A). Add about 60ml THF and mix it with a glass rod while heating at

40 degree C on a water bath. Leave it under gentle heating and stirring for 5 minutes, then filter it through two preweighed dry filter layers (glass micro fibre 934-AM Watman) using a Buchner funnel. Wash with fresh THF until colorless filtrate is observed. Place the filter papers and the solids in the oven (105 degree C) and leave it for 10 minutes. After drying, place it in a desiccator to cool before weighing to the nearest. Divide the weight of filter cake to the weight of sample and multiply by 100 to obtain the THF insoluble in percent weight.

VACUUM DISTILLATION

The apparatus is shown schematically in Figure I. The distillation is done in two stages: 20-40mm hex vacuum to remove a fraction which distills before 275 degree C 0.2 to 1mm Hg vacuum to obtain all the other fractions: 275 - 350 degree C 350 - 407 degree C 407 - 450 degree C - 450 - 525 degree C

PROCEDURE

Weigh 50-60gr of sample into the previously tared vacuum distillation flask. Seal the flask onto the apparatus using hi- temp-vac lubricant. Surround the traps with the proper coolant, start the vacuum and observe the flask content for signs of foaming. The pressure on the distillation assembly shall be lowered gradually to deaerate and minimize the foaming. When the equipment is air tight, apply a small flame into the flask. Adjust the burner so that the first drop of condensate will be collected after 10-15 minutes, at which point the temperature and pressure are noted and recorded. After the first fraction (275 degree C) is collected, the distillation is stopped, the U-tube manometer replaced by the McLeod gauge and the water condenser is replaced by a vacuum connecting tube to prevent the plugging of the line by the higher boiling fractions which tend to solidify at room temperature.

As soon as vapor or refluxing liquid appears at the joint between the flask and the column, adjust the rate of heating so that the distillation is recovered at a constant rate (approx. 2ml/min.). Continue the distillation until either the maximum temperature (525 degree C equivalent) is reached, or the cracking occurs (sudden rise in pressure or excessive smoking) in which case note the temperature at which cracking occurs.

After a distillation is completed, the apparatus should be allowed to cool for about 10 minutes before admitting air into it in order to prevent the possibility of vapor self-ignition. Use liquid nitrogen in the vacuum flask surrounding the glass trap.

Weigh the fractions collected and the residue, calculate the mass balance and store all fractions in sealed containers at -17 degree C for further analysis.

LABORATORY VACUUM DISTILLATION UNIT